

Retention of mercury by low-cost sorbents: Influence of flue gas composition and fly ash occurrence

A. Fuente-Cuesta, M.A. Lopez-Anton^{*}, M. Diaz-Somoano, M.R. Martínez-Tarazona

Instituto Nacional del Carbón (CSIC). C/ Francisco Pintado Fe N° 26, 33011, Oviedo,
Spain

**Corresponding author*

Phone: +34 985119090

Fax: +34 985297662

e-mail: marian@incar.csic.es

Abstract

The present study employs chars obtained from the gasification of different types of biomass as low cost sorbents of mercury at laboratory scale. The influence of gas composition and fly ash occurrence on mercury retention and oxidation by char samples was evaluated. Chars obtained from a mixture of paper and plastic waste showed mercury retention capacities similar to those obtained with a commercial activated carbon. Homogeneous mercury oxidation was mainly promoted by NO_2 and, to a certain extent, by $\text{SO}_2 + \text{O}_2$. The highest heterogeneous mercury oxidation was observed in the chars with the highest mercury retention capacity suggesting that the sorption process also involves the capture of oxidized mercury species. The presence of fly ash particles clearly influenced heterogeneous oxidation but did not affect mercury retention by the char sorbents.

Keywords: mercury; oxidation; char; biomass; fly ash

1. Introduction

On 2011 the USEPA (United States Environmental Protection Agency) proposed the first national standard to reduce mercury and other toxic air pollutants from coal and oil-fired power plants [1]. This final rule came into effect on April 16, 2012 [2]. At the same time other countries are also making considerable efforts to reduce mercury emissions and establish new legislation [3]. For example, the European Union (E.U.) has already made progress in addressing the global challenges posed by mercury by having it listed for consideration in the assessment and management of ambient air quality, under the European Commission's Air Quality Framework Directive (Council Directive 96/62/EC). In 2005 the European Commission launched the E.U.'s mercury strategy which explains the E.U. position concerning the international discussion on mercury [4]. There is obviously a growing demand for the development of low-cost mercury removal techniques that can be implanted in coal combustion plants.

It is difficult to define the best technique for mercury capture because there are many factors to consider such as the configuration of the air pollution control devices used in the power plants, the type of coal burned and so on [5]. Although different methods are being investigated [6-9], the injection of powdered activated carbon into the duct upstream of the particulate control device is the most developed technology for mercury capture. However, a notable drawback to the use of activated carbon for mercury capture in power plant flue gas is the annual operating cost. Activated carbons are expensive, ranging in price from \$500 to \$3,000 per ton. The annual cost of using activated carbon for mercury removal at a typical 500-MW_e coal-burning power plant is estimated to be around five million dollars [10]. Therefore, the use of low-cost sorbents, such as the ones proposed in this work (i.e. gasification chars), which are residues of the

process itself, could be an attractive alternative to activated carbons for direct injection into power plants.

Gasification char is the finest component of the gasifier slag. Apart from having the advantage of high carbon content and good textural characteristics, some char samples contain a large amount of chloride which improves their performance as sorbents for mercury retention. Activated coal chars have been tested for the retention of mercury in a number of studies [11-13]. However, the main benefit of using char residue from gasification is that it does not have to be pretreated, which reduces the cost. Fuente-Cuesta et al. [14] found that some chars resulting from the gasification of biomass, mainly from plastic-paper waste may exhibit a mercury retention capacity similar to that of a commercial activated carbon. However, the use of such sorbents is dependent on their behavior in real combustion atmospheres, and the influence of parameters, such as flue gas constituents (SO_2 , HCl , NO_x) and fly ashes on homogeneous and heterogeneous oxidation and on mercury retention capacity need to be evaluated.

It is well known that mercury in coal-fired power plants is found mainly as elemental mercury ($\text{Hg}^0(\text{g})$), oxidized mercury ($\text{Hg}^{2+}(\text{g})$) and particulate-associated mercury (Hg^{P}) and that, depending on its mode of occurrence, it can be retained by different types of air pollution control devices [15-16]. Gas composition plays a very important role in mercury speciation. As the combustion gases are cooled, homogenous reactions (gas-gas) [17-19] and heterogeneous interactions with fly ashes (gas-solid) may occur affecting the mercury speciation and retention [20-22]. Several studies have already confirmed the influence of fly ashes on retention and speciation of mercury [22-24] but heterogeneous reactions between the mercury and the solid sorbents may also occur and therefore need to be studied.

With the ultimate objective of developing low-cost sorbents for direct injection into power plants, the aim of this study was to evaluate the oxidation and retention of mercury by a series of biomass gasification chars taking into consideration the influence of gas composition and possible interactions with the fly ashes that may be present in the gases.

2. Experimental

Eight previously characterized [14] biomass gasification chars (SH, PL, CW, WW1-2 and PW1-3) of different origin were used in this study as mercury sorbents. The chars SH, PL, CW, WW and PW were the sub-product of the gasification of sunflower husks, poultry litter, clean wood pellets, wood waste and a mixture of paper and plastic waste, respectively. The chars were obtained from a pilot gasification plant of 500 kW with a circulated fluidized bed (CFB) gasifier BIVKIN.

The experimental device employed to retain mercury in the char samples at laboratory scale is shown in Figure 1. The sorbent bed was prepared by mixing 20 mg of char with 60 mg of sand. This mixture was then placed inside a glass reactor. Blank experiments were carried out using only sand as sorbent bed. The experiments were carried out at 150°C. This temperature is inside the temperature range for the gas at the sorbent injection point upstream of particulate matter control devices [25]. Elemental mercury in gas phase obtained from a permeation tube was passed through the sorbent bed at a flow rate of 0.5 L min⁻¹. The mercury concentration in gas phase was approximately 100 µg m⁻³. A synthetic gas mixture consisting of 5% O₂, 1300 mg Nm⁻³ SO₂, 500 mg Nm⁻³ NO₂, 20.3 mg Nm⁻³ HCl and N₂ was passed through the reactor. To evaluate the effect of the gas composition on mercury retention, the results were compared with those obtained in a N₂ atmosphere. In general, the duration of the

mercury retention experiments was the time needed for the samples to reach maximum retention capacity and this varied depending on the type of char employed. The mercury not retained in the sorbents was measured using a continuous mercury monitor (VM-3000). The mercury content after the retention experiments was determined by means of AMA equipment.

The possible oxidation of mercury was evaluated by capturing the oxidized mercury in an adsorption resin before the elemental continuous mercury analyzer. For this purpose a Dowex[®] 1x8 chloride form resin was placed at the exit of the reactor (Figure 1). Dowex is an ion exchanger designed specifically for the selective extraction of mercury(II) species. This material allowed the simultaneous evaluation of mercury retention and oxidation over long periods of experimentation. The resin was treated before the experiments with a mixture of HCl:H₂O (1:1) at 90°C for 30 minutes and then filtered and dried. It was analyzed at the end of the retention experiments by means of AMA to determine the amount of mercury that had been oxidized. In addition to the N₂ and the complete atmosphere used in the mercury retention experiments, atmospheres consisting of O₂, SO₂, NO₂ or HCl in N₂ or any combination of these compounds in identical proportions were passed through the experimental device to evaluate the influence of each gas upon mercury speciation.

A fly ash denoted as CTL was selected in this work to evaluate the effect of the fly ash particles on the char samples used as mercury sorbents. CTL was obtained from a pulverized coal combustion (PCC) power plant whose main combustible component was bituminous coal. The retention and oxidation experiments were carried out as described above except that in this case the sorbent bed was prepared by mixing 20 mg of char with 20 mg of fly ash and 40 mg of sand.

3. Results and discussion

The results of this study have been divided into the following parts to facilitate discussion: (i) the retention of mercury by the raw biomass gasification chars in an inert and simulated coal combustion atmosphere; (ii) the homogeneous and heterogeneous oxidation of mercury and (iii) the influence of fly ashes on the oxidation and retention of mercury by the char samples.

3.1. Mercury retention

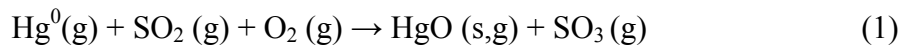
In a previous work [14] raw gasification chars from different biomasses were studied to evaluate their mercury adsorption capacity in relation to their properties. Different mercury retention capacities were obtained depending on the type of biomass char. No relationship was found between mercury retention and the majority of the inorganic components of the chars. A higher mercury capture was observed only in the samples with high aluminium and chloride contents. The surface oxygen groups appeared to have no effect upon mercury capture, according to programmed temperature desorption analysis, probably because of interference by the inorganic components. It was found that an increase in the surface area of the char samples enhances mercury capture. Although there was no linear relation between mercury captured and unburned carbon content (represented as lost of ignition, LOI), examination of the retention by each group of chars from the same type of biomass (e.g. plastic-paper or wood) showed that retention increased when the LOI increased. Therefore, apart from the characteristics of each char, the influence of other parameters such as the composition of the flue gases during coal combustion must be taken into account in order to explain the different mercury retention capacities obtained for each type of char. To assess the influence of the flue gases the results of mercury retention were compared in an inert and a synthetic gas mixture from coal combustion (Table 1). The confidence limit of the

results, established as the relative standard deviation, is <15 % except for the SH and PL chars which showed a standard deviation of approximately 25%. It should be mentioned that the samples from sunflower husks and poultry litter were very heterogeneous in size and composition. In fact, it was necessary to increase the number of replicates for each experiment with these samples, since the deviation in the results was higher than for the rest of the samples. Table 1 also shows the results obtained for a commercial activated carbon impregnated with sulphur (Filtracarb D47/7+S) in the two atmospheres. The possible retention of mercury in the sorbent bed was evaluated by using inert material (sand) only, as sorbent. Mercury was not retained in this material in the experimental conditions of this study. As was pointed out in a previous work [14], the chars from plastic and paper waste showed a similar mercury retention capacity to that of activated carbon. In this study, mercury retention was consistently higher in a simulated coal combustion atmosphere than in a nitrogen atmosphere, especially in the case of the SH char (Table 1). In the simulated combustion atmosphere mercury retention capacity of the chars can be represented as follows: PW > SH > PL ~ WW > CW. However this order is not preserved in the nitrogen atmosphere as there is a strong increase in mercury retention by the sunflower husks char in the combustion atmosphere (Table 1). The results seem to indicate that new mercury compounds were formed as a result of homogeneous oxidation which facilitated mercury retention in the char samples. Alternatively certain characteristics inherent in the chars may have led to interactions with the gases (heterogeneous oxidation) and favoured mercury retention.

3.2. Mercury oxidation

3.2.1. Homogeneous oxidation

It is known that mercury oxidation is promoted by the presence of NO_x, SO₂ and HCl. However, the extent of this oxidation depends on their relative proportions and concentrations and kinetic limitations, among other factors. Figure 2 shows the results obtained for Hg⁰ and Hg²⁺ for each of the gas compositions studied. The results indicate that no homogeneous oxidation reactions occurred in the single gases O₂, SO₂ and HCl under the experimental conditions of this study. However, in the NO₂ atmosphere approximately 11% of Hg²⁺ was formed. When O₂ was present with SO₂ and NO₂, there was 4 and 24% of oxidation, respectively. This would suggest that O₂ has a synergistic effect on mercury oxidation not only thorough SO₂ which agrees with results of a previous study [20], but also via NO₂. When all the components were mixed (O₂ + NO₂ + SO₂ + HCl + N₂) the mercury oxidation percentage was similar (15%) to that obtained with O₂ + NO₂ + N₂ (24%) (Figure 2). Thus it can be deduced that SO₂ mixed with O₂ and mainly NO₂ with or without O₂ are the main gas combinations responsible for the homogeneous oxidation of mercury. The gas species that might be present is HgO which would form through the reactions (1-2).



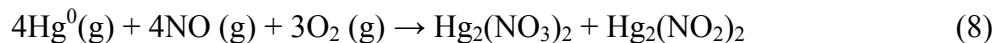
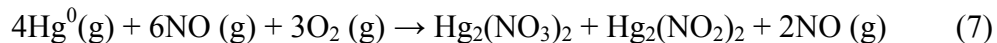
NO₂(g) would start to decompose at 150°C (reaction 3). Although this reaction is sometimes slow, adding O₂ to the gas stream would tend to drive reaction 3 to the left and favour mercury oxidation via reaction 2. This would explain the higher mercury oxidation under the NO₂ + O₂ atmosphere compared to the NO₂ atmosphere (Figure 2).



3.2.2. Heterogeneous oxidation

Heterogeneous oxidation was evaluated using sorbent beds made up of char. Figures 3-4 show the percentages of Hg^{2+} and Hg^0 in the outlet gas and the percentage of mercury retained in the char which is referred to as particulate mercury (Hg^{P}). The atmospheres employed were N_2 (Figure 3) and the mixture $\text{O}_2+\text{NO}_2+\text{SO}_2+\text{HCl}+\text{N}_2$ (Figure 4). In the N_2 atmosphere, where there was no homogeneous oxidation (Figure 2), approximately 4% of Hg^{2+} was obtained in all the char samples. Therefore, the char samples participated slightly in the oxidation of mercury in the inert atmosphere. In the atmosphere containing $\text{O}_2+\text{NO}_2+\text{SO}_2+\text{HCl}+\text{N}_2$ the amount of Hg^{2+} collected at the exit of the reactor was similar (~20%) to that estimated for the homogeneous oxidation of the chars from sunflower husks, poultry litter and clean wood pellets (Figures 2 and 4). This shows that either heterogeneous oxidation hardly occurred in this atmosphere or that all of the Hg^{2+} produced from heterogeneous oxidation was retained in these chars. However, approximately 35% of the mercury in gas phase was in oxidised form in the case of chars from wood waste, whereas it was 40-60% in the case of the chars from plastic and paper (Figures 2 and 4), indicating that heterogeneous oxidation did occur. With the exception of char SH, the oxidation of mercury was higher in the chars with a higher mercury retention capacity in the simulated coal combustion atmosphere (Table 1, Figure 4). In addition to reactions (1-3) which explain the homogenous oxidation, when char is present it may act as support for the formation of HgSO_4 in a $\text{SO}_2 + \text{O}_2$ atmosphere (reaction 4) [20] and/or different mercury nitrites and nitrates in NO_2 atmospheres (reactions 5-8) [26-27]. Therefore in an atmosphere containing $\text{O}_2+\text{NO}_2+\text{SO}_2+\text{HCl}+\text{N}_2$ all of the above reactions may take place. We must also remember that the mercury experiments in this work were performed at 150°C and most mercury nitrites and nitrates are unstable over 100-200°C [26,28].





Mercury sulphate and mercury nitrites and nitrates should be in the form of a solid deposited on the char. This would explain the larger amount of mercury retained by the char samples in the $\text{O}_2 + \text{NO}_2 + \text{SO}_2 + \text{HCl} + \text{N}_2$ atmosphere compared to the in N_2 atmosphere (Table 1). Retention in the 5-gas atmosphere is especially high in the SH char from sunflower husks, which is the char that has the highest unburned carbon content (LOI=78%). The LOI values ranged from 25 to 55% for the others [14]. This suggests that the carbon particles may be favouring the oxidation of mercury through reactions 4-8. Furthermore, in the presence of solid species, $\text{HgO}(\text{g})$ may remain in gas phase, because mercury sulphate and mercury nitrites and nitrates may prevent it from binding itself to the char [20]. This would explain the increase in the amount of Hg^{2+} emitted in the $\text{O}_2 + \text{NO}_2 + \text{SO}_2 + \text{HCl} + \text{N}_2$ gas mixture, in the presence of some chars (Figure 4). The oxidation values for SH were similar to those obtained during homogeneous oxidation (Figures 2 and 4) but mercury retention in the gas mixture was much higher. This indicates that the Hg^{2+} species were retained in the char with the highest carbon particle content. Consequently, oxidation followed by retention cannot be ruled out, since retention of mercury in the chars (Hg^{P}) would result from both homogeneous and heterogeneous oxidation.

3.3. Influence of fly ash

To evaluate the influence of the fly ash particles on the oxidation and retention of mercury by chars a series of experiments were carried using a sorbent bed of char and fly ash (CTL). The goal of this study was to determine the influence of the ash particles present in the flue gas on mercury retention and oxidation. Table 2 shows the mercury retention capacities in the inert and the $O_2+NO_2+SO_2+HCl+N_2$ atmospheres using the sorbent char+fly ash. These tests were carried out using a representative sample of each type of chars. Considering that the standard deviation of the results was between 10 and 20% for all the mixtures of char+CTL with the exception of the SH+CTL and PL+CTL mixtures whose standard deviation was approximately 30%, the results may be considered similar to those obtained when only the char was used as sorbent (Table 1). It can be concluded, therefore, that the mercury retention capacity of the chars is not affected by the presence of fly ash particles in the experimental conditions of this study (Table 1). A slight increase in the retention capacity of the SH and PL chars can be observed in the atmosphere containing $O_2+NO_2+SO_2+HCl+N_2$ in the presence of CTL (Table 2) but, as already mentioned, these chars are very heterogeneous materials and it was difficult to obtain a representative sample for testing.

The influence of fly ash on heterogeneous mercury oxidation was also studied in the $O_2+NO_2+SO_2+HCl+N_2$ atmosphere (Figure 5). Approximately 60% of Hg^{2+} was detected in the outlet gas phase with the fly ash CTL in the bed. Given that homogeneous oxidation was ~20% (Figure 2), this shows that CTL promotes mercury oxidation. Similar percentages of Hg^{2+} were found in the mixtures of char+fly ash (Figure 5). The SH, PL, CW, WW2 and PW1 chars favoured mercury oxidation slightly (Figure 4) confirming that the percentages of Hg^{2+} observed in char+CTL sorbent are due mainly to homogeneous oxidation and to heterogeneous oxidation on the fly ash. It

can be observed that the highest errors are again occurred in the most heterogeneous samples (Figure 5). The results for mercury oxidation when fly ash was present are consistent with the fact that no significant variations were observed in the mercury retention capacities between the char and char+fly ash samples (Table 2).

4. Conclusions

The highest mercury retention capacities were obtained with the chars from plastic and paper waste and these were similar to those achieved with a commercial activated carbon impregnated with sulphur. All the char samples studied showed higher mercury retention capacities in a simulated coal combustion atmosphere than in an inert atmosphere. SO₂ mixed with O₂ and mainly NO₂ with/without O₂ were the main factors responsible for the homogeneous oxidation of mercury. In general, the chars with the highest mercury retention showed the highest mercury heterogeneous oxidation suggesting that the formation of mercury oxidized species such as mercury sulphate and mercury nitrites and nitrates occurs with homogeneous oxidation in some chars. Mercury retention by the char samples was not affected by the fly ash particles. However, these particles had the effect of increasing the proportion of oxidized mercury.

Acknowledgments

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Table 1. Mercury retention capacities in N₂ and simulated coal combustion atmospheres in the char samples and the commercial activated carbon.

Sample	Hg retained ($\mu\text{g}\cdot\text{g}^{-1}$)	
	N ₂	Combustion
SH	<1	120±30
PL	1.1±0.3	36±10
CW	<1	6.0±1
WW1	2.7±0.4	33±5
WW2	2.7±0.3	31±4
PW1	135±24	172±31
PW2	110±12	164±18
PW3	65±9	78±11
Filtracarb	145±32	227±50

Table 2. Mercury retention capacities in N₂ and simulated coal combustion atmospheres in the char samples in the presence of fly ash.

Sample	Hg retained ($\mu\text{g}\cdot\text{g}^{-1}$)	
	N ₂	Combustion
CTL	2.2±0.4	10±2
SH+CTL	1.5±0.6	155±35
PL+CTL	1.7±0.5	38±12
CW+CTL	1.5±0.3	19±4
WW1+CTL	4.3±0.7	33±6
PW1+CTL	146±15	165±17

Figure captions

Figure 1. Schematic diagram of the experimental device.

Figure 2. Percentages of Hg^0 and Hg^{2+} in the different atmospheres studied.

Figure 3. Mercury speciation in an N_2 atmosphere in the presence of different char samples.

Figure 4. Mercury speciation in a simulated coal combustion atmosphere in the presence of different char samples.

Figure 5. Mercury speciation in a simulated coal combustion atmosphere in the presence of different char samples and fly ash.

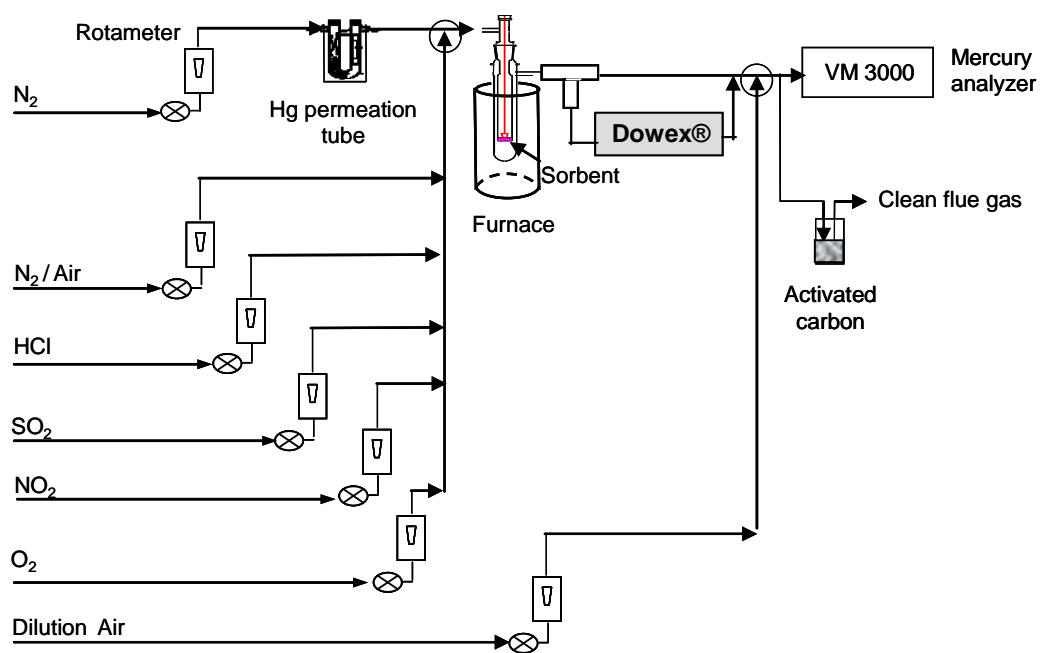


Figure 1

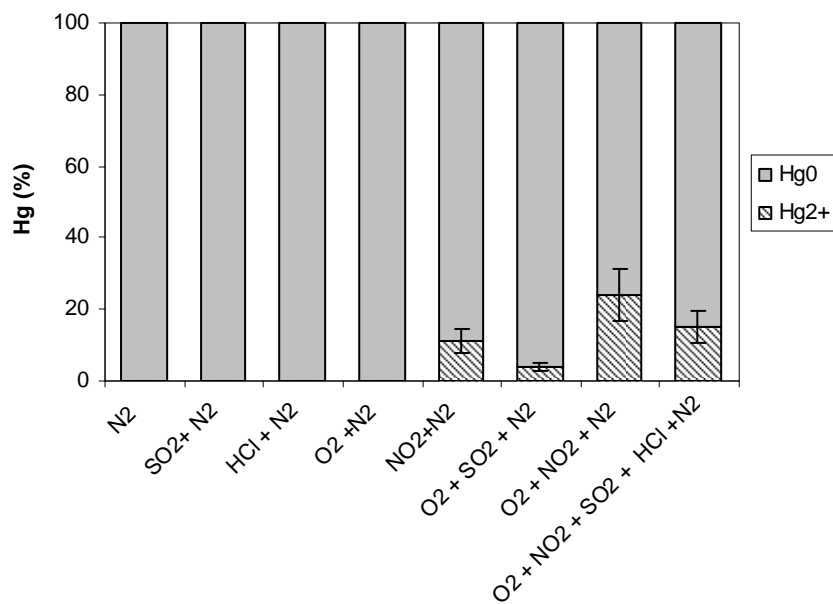


Figure 2

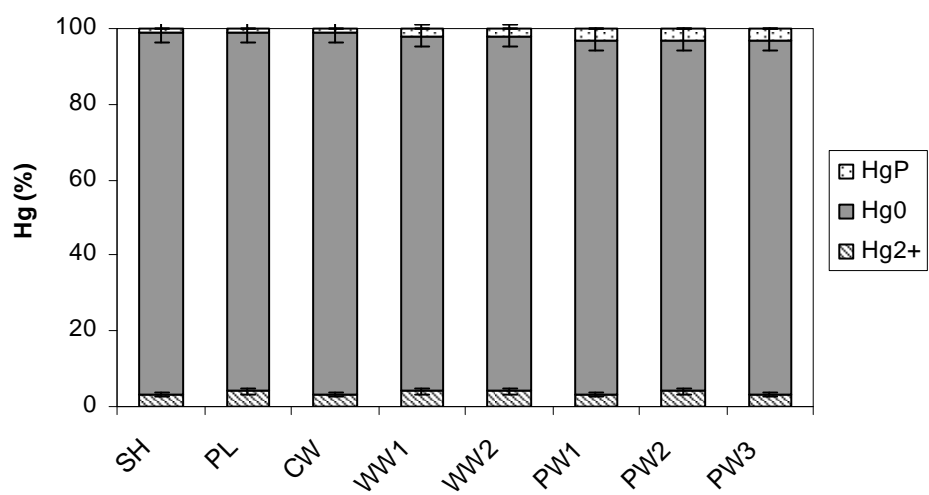


Figure 3.

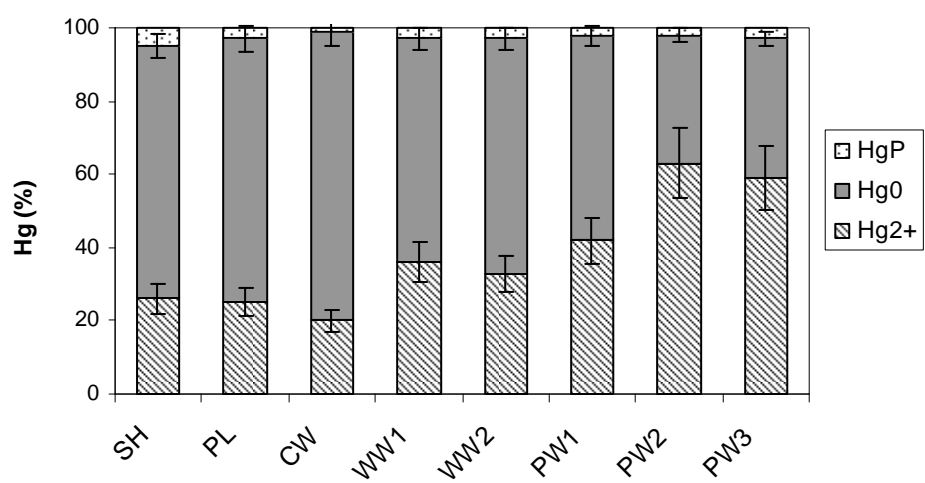


Figure 4

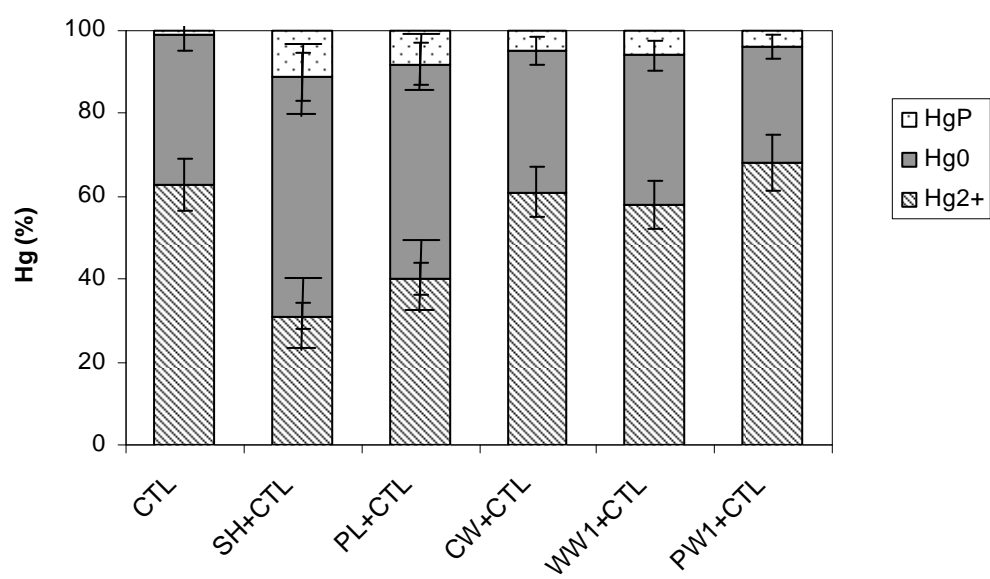


Figure 5